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Evaluation of Lithium Hydroxide for Alkalinity Control of Boiler Water Softened With EDTA Chelating Agent

Assignment 73 120
MEL R&D Phase Report 209/65
June 1965

By
E. H. Bolander

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Machinery Systems Division

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ABSTRACT

An experimental boiler water treatment for 1200 psi boilers, using lithium hydroxide (LiOH) and the disodium salt of ethylenediaminetetraacetic acid, was evaluated in a series of 30-day studies in model boilers.

This combination was found preferable to the present method (low-phosphate control treatment) or to a modification of the present method in which LiOH was substituted for NaOH for pH control (previously reported).

When LiOH and NaOH were used independently, without other additives, LiOH was shown, by metallographic examination, to have superior corrosion inhibitive properties. When LiOH was combined with Na_2EDTA and dilute seawater (0.5 epm chloride), slight corrosion was detected, but inhibition was superior to that exhibited by NaOH alone. This work is continuing.

ADMINISTRATIVE INFORMATION

The work reported herein was performed under MEL Assignment 73 120, Sub-project S-R007 08 08, Task 0613. The project was authorized by Bureau of Ships letter R007-08-08, serial 634A-226 of 22 May 1963. A proposal for additional work on this project was submitted to the Bureau of Ships by MEL letter NP/11330(833) of 26 May 1964.

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EVALUATION OF LITHIUM HYDROXIDE FOR ALKALINITY CONTROL OF BOILER WATER SOFTENED WITH EDTA CHELATING AGENT

1.0 INTRODUCTION

The use of lithium hydroxide (LiOH) instead of sodium hydroxide (NaOH) in boiler water has been demonstrated in this Laboratory[1] and by the Naval Research Laboratory[2] to significantly reduce corrosion of boiler tube metal in contact with the treated water. When LiOH was used in place of NaOH for pH control with the conventional Navy low-phosphate control treatment for 1200 psi* boilers, however, the advantage of minimized corrosion was diminished by the rapid consumption of phosphate treating chemicals, excessively high pH values, and, most seriously, by extensive deposits of lithium phosphate (Li_3PO_4) on heat transfer surfaces. Such deposits would cause tube failures due to overheating of the metal.

This report discusses the evaluation of an alternate method for softening of boiler water which permits the use of LiOH for pH control. The treatment makes use of an organic chelating agent instead of the phosphates presently used.

The disodium salt of ethylenediaminetetraacetic acid (Na_2EDTA) was used as the chelating agent throughout these studies. This chemical has been widely used as an analytical reagent[3] and more recently has been produced commercially for chemical cleaning and water softening purposes. References 4 through 9 report its successful use as a softening agent in high-pressure, power plant utility boilers. Reference 10 discusses its use in formulations for in-service boiler waterside cleaners. The cost of the chelating agent is considerably higher than that for the conventional phosphate treatment, but its use is considered economically feasible when resulting elimination

[1]Superscripts refer to similarly numbered entries in the Technical References at the beginning of this report.

*Abbreviations used in this text are from the GPO Style Manual, 1959, unless otherwise noted.

of periodic chemical cleaning of boilers and reduced outages from tube failures are taken into account.

A base line for comparison of the corrosivity of the EDTA treatment was established by operating the test boilers with solutions of NaOH and LiOH only and observing comparative effects of metal corrosion.

2.0 EVALUATION

The evaluation was conducted in a series of 30-day tests, using one of the electrically heated model boilers described previously.[1] Because of the relatively low heat flux (ca 20,000 Btu per (sq ft)(hr)) in the model boiler, the chemicals (LiOH and NaOH) were not expected to concentrate in sufficient quantities to cause embrittlement of the tube surface. An embrittlement detector, designed to concentrate boiler water solids in a crevice, was used to determine the effect of such concentrations on highly stressed mild steel specimens. The relative corrosivity of the boiler water solutions was evaluated by metallographic examination of the embrittlement detector specimens and boiler tubes.

2.1 Equipment.

2.1.1 Model Boiler. The boilers were the Nalco A type in which a central vertical cylinder serves a combined steam drum, downcomer, and water drum. A V-shaped sidearm loop encloses an electrical heating element (providing 6800 Btu per hr) which is housed in a removable mild steel tube. This tube serves as a 20,000 Btu per (sq ft)(hr) steam generator and permits inspection of waterside heat transfer surfaces after the boiler is drained.

2.1.2 Embrittlement Detector. The embrittlement detectors were the U. S. Bureau of Mines type described in reference 11. These are highly stressed mild steel specimens which are exposed to concentrated boiler water solids deposited by flashing steam through an orifice onto the stressed area. The detectors thus provide the necessary mechanical conditions for susceptibility to stress corrosion cracking.

2.1.3 Modifications. In addition to the embrittlement detectors used to detect corrosion resulting from concentration of boiler water solids, the heating elements (specimen boiler tubes) were

modified to form inspection sites on which dissolved solids would concentrate preferentially. This was done to demonstrate reactions on metal surfaces resulting from the treatment chemicals. The modifications were as follows:

- Insertion of removable specimens with underlying void areas.

- Cutting of a circumferential groove (1/4 inch wide and 1/8 inch deep) around the specimen tube.

- Drilling of a series of holes of varying diameters (1/32 to 1/8 inch in 1/32-inch increments) to a depth of 1/8 inch axially along the heated surface of the specimen tube.

To provide maximum representation of the heated surface, four series of holes and the dovetailed slots for the removable specimens were located at 90° positions (top, bottom, and sides) around the circumference of the heated tubes. The outside diameter of the tubes was increased by 1/4 inch from the original design to provide a minimum wall thickness consistent with safety requirements (ASME boiler code). Figures 1 and 2 are photographs of the experimental tubes.

Initial attempts were made to ensure uniform rates of heat transfer from the stainless steel resistance heaters through the mild steel specimen tubes by use of a liquid metal (molten tin) heat transfer medium in the void between the heater sheath and sides of the specimen tube in contact with the heater described in paragraph 2.1.1. This proved to be unsatisfactory because of a high loss of heaters through burnout. These failures were caused either by hot spots from air voids in the molten tin, or by attack, by the tin, of a soldered seam at the base of the heating elements or by poor wetting of the heater by the tin. These tests were then made without a liquid heat transfer medium, using drilled heater receptacle holes as specified by the heater manufacturer.

2.2 <u>Experimental Procedures</u>. The model boilers were cleaned with inhibited hydrochloric acid and flushed with distilled water. They were passivated with a sodium nitrite solution, drained, flushed with distilled water again, drained, and dried with nitrogen.

2.2.1 Test Conditions.

Run 1: The embrittlement detector and boiler tube were exposed to 1200 psig boiler water containing only NaOH. Sufficient alkali was present to obtain a pH of 10.8. (This value is within the prescribed range (10.4-11.0) for naval boiler water in 1200 psi boilers.)

Run 2: Repeat of Run 1 using LiOH instead of NaOH for pH control.

Run 3: Repeat of Run 2 using boiler water containing 0.5 equivalents per million (epm) chloride ion (Cl^-) added as synthetic seawater. (This was the maximum allowable chloride content for shipboard feedwater.)* This concentration of dilute seawater contains 6.28 ppm hardness expressed as calcium carbonate and results from the combined calcium and magnesium content of the seawater. Sufficient disodium EDTA (0.045 gram per liter) was added to the water to chelate twice this hardness value. The excess chelating agent was used to compensate for possible partial decomposition and reaction with metal ion in the boiler water.

2.2.2 Test Procedure. The boilers were filled to operating levels with feedwater containing the treatment chemicals. The feedwater, as well as the initial charge of boiler water, was treated with the chemicals because of the limited capacity of the model boilers. This technique prevented extreme dilution of dissolved chemicals in the boiler water when part of it was withdrawn (for analysis or blowdown), since treated water was available for replenishment, whereas the treated water would normally be replaced with distilled water. The feedwater was deaerated by bubbling nitrogen through the solutions and maintaining a slight pressure of nitrogen on the feed tank at all times. A chemical injector, located between the feed pump and the boiler, provided a means of fortifying weak solutions resulting from reactions of treatment chemicals which formed insoluble compounds. The electrical resistance heaters were then energized and the water was heated until the pressure reached 1200 ±50 psig. A minimum steam leak was directed against the stressed specimen in

*Recently reduced to 0.2 epm Cl^-.

the embrittlement detector. The minimum leak rate was considered to be reached when a fogged area was observed on a mirrored metal surface held near the detector orifice. Thereafter, the leak was checked daily and the leak rate adjusted when necessary.

During each exposure period (30 days), boiler water samples were withdrawn daily and analyzed for pH and alkalinity. During Run 3 (LiOH + EDTA treatment), an additional test was performed daily for either hardness or residual (unreacted) EDTA. These additional tests were made by complexiometric titrations using the azo dye Eriochrome Black T as an indicator. Hardness or EDTA deficiency was determined by titration of 100-ml samples with standard (0.005 N) EDTA solution (1 ml = 5 ppm $CaCO_3$). Residual EDTA was determined by titration with 0.005 N magnesium sulfate solution (1 ml = 18.6 ppm Na_2EDTA). The reagents and details are described in reference 4. On the basis of results of these analyses, either additional chemicals were injected or the boilers were blown down. To prevent concentration of solutions within the boiler, no steam was removed during the test period.

2.2.3 Inspection. After 30 days of exposure, the boilers were drained and the boiler tube and embrittlement specimens were removed, examined, and photographed. Both boiler tube and embrittlement specimens were sectioned and examined under a microscope to expose evidence of surface corrosion. Photomicrographs were made of typical surface oxidation or subsurface attack.

3.0 RESULTS

The test conditions for each run and the results of the visual examinations of the specimens after each run are summarized in the following tabulation. (See page 7.)

4.0 DISCUSSION OF RESULTS

Run 1: NaOH Treatment, 30 Days. No difficulties were encountered in maintaining pH values within the specified range (10.4-11.0). Actual pH values ranged from 10.8 to 11.0. Daily alkalinity analysis showed that the concentration of hydroxyl ions ranged between 0.4 and 1.5 epm. Other than the withdrawal of analytical samples (approximately 300 ml), no blowdown was required. No injections of NaOH, other than that present in the feedwater, was required.

Both the embrittlement specimen and the boiler tube were removed for examination. After 30 days of exposure, the tube was coated with a magnetite film and was free from foreign deposits. No cracks were detected in the embrittlement specimen when examined under low power magnification (60X). Additional stressing of the specimen by further bending failed to reveal any cracks that may have formed but were too fine to be visible without additional stressing of the surface. The embrittlement specimen and the boiler tube were sectioned to expose surface films and underlying metal. Metallographic examination of these specimens at 250X showed significant surface oxidation. Most of the corrosion occurred in the small diameter holes drilled in the tube walls. Of the several diameter holes used, the largest (1/8-inch diameter) showed the most significant attack. Exhibit "A," Figure 1 shows the tube and embrittlement specimen from this run. Figure 3 shows an area at the base of a hole where severe pitting was noted.

Run 2: LiOH Treatment, 30 Days. Some difficulty was experienced in maintaining the pH within the desired range. On the ninth day, the pH reached the maximum allowable limit (11.0) and required boiler blowdown. Daily alkalinity analysis showed that the hydroxyl ion concentration ranged between 1.3 and 1.7 epm. These comparatively high results may have been caused by atmospheric carbon dioxide which had been adsorbed in the LiOH stock solution with which the feedwater was treated. If this carbon dioxide were later released in the steaming boiler, increased pH and alkalinity values would result.

Visual examination of the embrittlement specimen and boiler tube showed no unusual effects. Exhibit "B," Figure 1 shows the tube and embrittlement specimen after 30 days of exposure from this run. No cracks were found in the embrittlement specimen. Metallographic examination (250X) of these specimens after sectioning revealed a relatively thin, uniform oxide film with virtually no subsurface attack. A representative section is shown in Figure 4.

Run 3: LiOH + Seawater + EDTA, 30 Days. The pH and alkalinity values obtained during this run were somewhat higher than those observed in Run 1. Blowdown was required on five occasions when pH values exceeded the prescribed maximum limit of 11.0.

Test Results

Run No.	Feedwater Composition	Additional Boiler Water Controls Required During Exposure	Observations After Exposure: Boiler Tube	Observations After Exposure: Embrittlement Specimen
1	Deionized water containing enough NaOH to obtain pH of 10.8.	None. Boiler water pH ranged from 10.8-11.0. Alkalinity ranged from 1.0-1.5 epm.	Visual inspection revealed only a normal black oxide film (Exhibit "A," Figure 1). Microscopic examination of sectioned tube showed relatively severe metal attack (Figure 3) most prevalent on walls and base of 1/8-inch-diameter holes.	No cracks detected. Relatively heavy and irregular surface oxidation but not as pronounced as that found in holes in boiler tube.
2	Deionized water with LiOH added to obtain pH of 10.8.	Approximately 30% of boiler capacity discharged to waste on 9th day when pH values exceeded 11.0. Alkalinity values ranged from 1.3-1.9 epm.	Appearance by visual examination similar to Run 1 (Exhibit "B," Figure 1). Microscopic examination revealed considerably less surface oxidation and no subsurface attack (Figure 4), as noted for Run 1.	No cracks detected. Some uniform surface oxidation but considerably less than that observed for Run 1.
3 and 3A	Deionized water with synthetic seawater added to obtain chloride concentration of 0.5 epm Cl^- and 6.3 ppm hardness as $CaCO_3$ + 45 ppm Na_2EDTA + LiOH to obtain pH of 10.8.	Boiler blown down on 5th day to reduce excessive pH value (>11.0). Hardness (0.5-1.0 ppm $CaCO_3$ detected on 3rd day when sufficient Na_2EDTA was in- [illegible] -tore [illegible] Mo[illegible] residu[illegible] ranged from 1.0-7.0 ppm Na_2EDTA, showing some decomposition of unreacted EDTA or reaction with dissolved corrosion products during the exposure.	Appearance by visual examination revealed exceptionally clean metal surfaces after 30 days of exposure (Figure 2). Exposure was then continued for a second 30-day period (Run 3A), after which microscopic examination of the sectioned specimen showed slight attack of metal surfaces but much less extensive than 30-day results obtained in Run 1 (Figure 5). Examination of a sample of the deposit scraped from the tube by X-ray diffraction showed lithium ferrite ($LiFe_5O_8$) to be the only identifiable constituent.	No cracks detected. Microscopic examination after 60 days showed slight irregular surface oxidation noted but considerably less pronounced than that seen on specimen from Run 1.

Hardness was detected on three occasions, ranging from 0.5 to 1.0 ppm (as $CaCO_3$). The boiler water was fortified at these times by injecting sufficient Na_2EDTA to restore the original excess of the chelating agent. During the remainder of the run, residual Na_2EDTA ranged from less than 1 to 7 ppm, indicating some decomposition of the free EDTA or reaction with dissolved corrosion products.

After 30 days of exposure, the specimens were removed and examined without magnification or further stressing of the embrittlement specimen. All surfaces appeared to be in excellent condition with no apparent corrosion. They were then reinstalled and exposed for a second 30-day period.

Run 3A: LiOH + Seawater + EDTA, 30 Days. The embrittlement specimen and heater tube from Run 3 were used for this run. A fresh feedwater solution was prepared with fresh LiOH to preclude the possibility of CO_2 being absorbed in the LiOH stock solution.

Although pH values rose to the prescribed maximum on the third day, no blowdown was required during this run to control pH. Except for the 3 days when the pH was 11, other values ranged from 10.6 to 10.9, with corresponding alkalinity concentrations ranging from 1.0 to 1.9 epm.

Figure 2 shows the appearance of the heater tube and embrittlement specimen after the first and second 30-day exposure periods.

Metallographic examination of the specimens sectioned after the second 30-day exposure showed metal surface oxidation to be uniform and similar to that found in Run 2. There was some slight subsurface attack, which is attributed to the combined effects of longer exposure and possible corrosive attack from sodium in the dilute seawater or treatment chemicals (di-sodium EDTA and sodium salts in seawater). A typical area is shown in Figure 5. The presence of chlorides in the diluted seawater may also have been responsible for the slightly more aggressive action in this run.

Examination of a sample of the deposit scraped from the surface of the boiler tube showed lithium ferrite ($LiFe_5O_8$) to be the only identifiable constituent.

5.0 DISCUSSION AND CONCLUSIONS

The results of these preliminary tests appear to confirm the hypothesis that the use of EDTA with LiOH would be beneficial in treating naval boiler water by minimizing corrosion and scaling of metal heat transfer surfaces. The problem of insulating deposit formation resulting from the use of LiOH with phosphate treating chemicals is eliminated. Further improvement may be gained by further reducing boiler water contamination by maintaining lower

feedwater chloride limits and the use of a more stable chelating agent or other water softening chemical.

The results of this evaluation demonstrate some significant attributes of the experimental boiler water treatment.

- These results complement those reported by NRL under static conditions (capsule tests) and show that treatment with LiOH results in less aggressive metal attack than similar treatment with NaOH.

- The combined indication of continuous reduction of EDTA above that required for softening and the excellent appearance of the specimen boiler tube after the first 30-day exposure indicate that some in-service cleaning is accomplished by use of the chelating agent.

- The problem of forming insulating deposits experienced with the substitution of LiOH for NaOH in the present Navy low-phosphate treatment, previously reported, is eliminated.

- The existence of slight corrosion with this treatment when exposed to an environment of sea-water contamination shows a necessity for further study in this area. Future tests will include experiments in which gross quantities of seawater are added to the boiler water to simulate shipboard conditions resulting from gross condenser leakage or a possible tactical situation which would require makeup of condensate return with seawater rather than evaporator distillate.

6.0 RECOMMENDATIONS

Recommendation of a full-scale trial in a shipboard boiler of the use of LiOH as a water treatment chemical is withheld pending results of current and planned future tests in the Nalco boilers.

7.0 FUTURE WORK

7.1 Tests are currently under way, using the Nalco boilers, to assess the use of the tetralithium salt of ethylenediaminetetraacetic acid (Li_4EDTA) for water softening. LiOH is used to control the pH. Results will be reported when the work is completed.

7.2 The effect of gross amounts of seawater, due to condenser leakage or operational requirements, will be studied in LiOH chelate systems.

7.3 The use of borates for water softening with LiOH for alkalinity control will also be investigated.

7.4 A desk study of the feasibility of using ion exchange resins for feedwater treatment has recently been completed. The report is being prepared.

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Exhibit "A"
NaOH, Run 1

Exhibit "B"
LiOH, Run 2

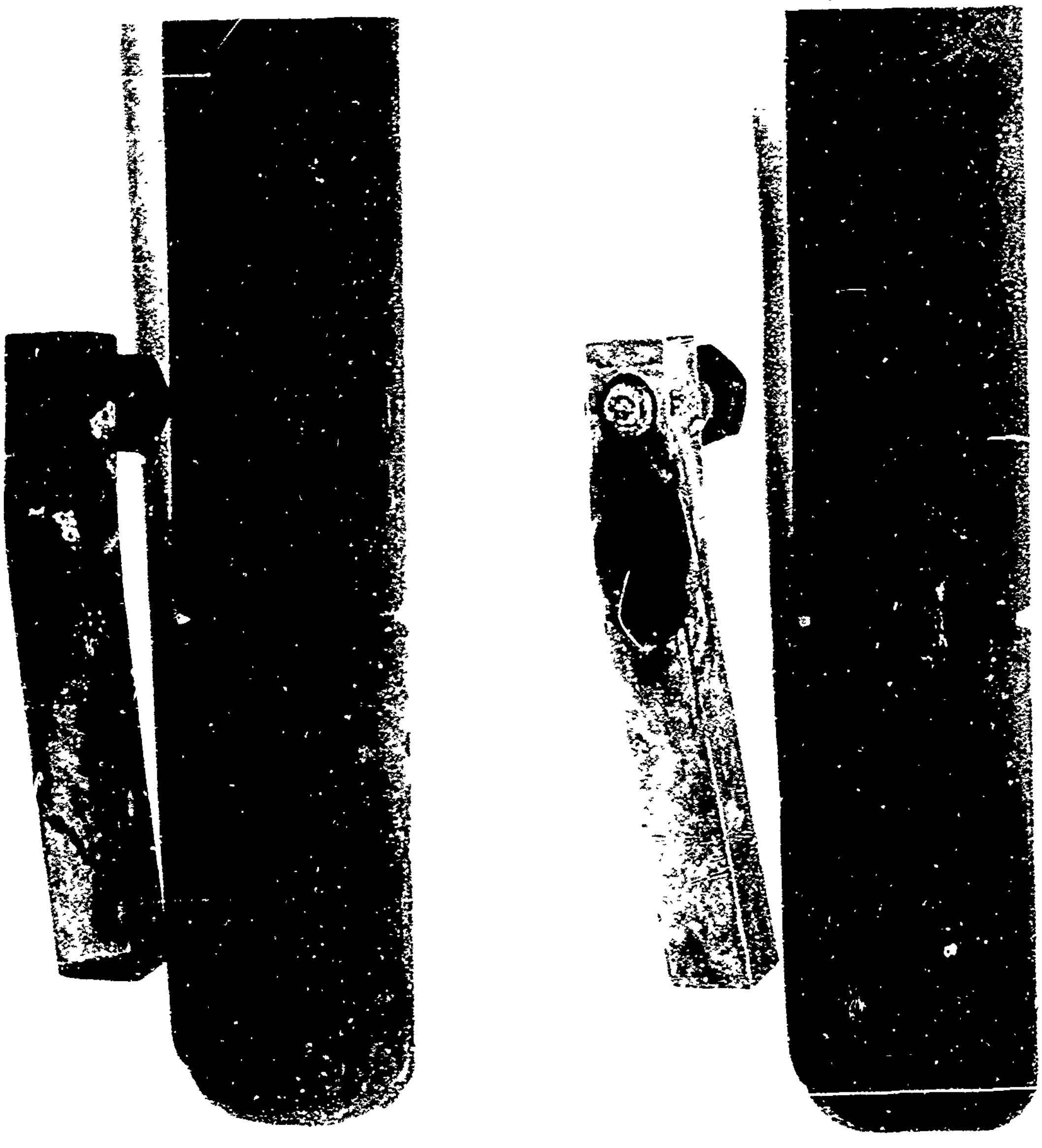

Figure 1
Boiler Tubes and Embrittlement Specimen After 30-Day Exposure to 1200 PSI Boiler Water Treated With Alkaline Chemicals Only (NaOH or LiOH to pH 10.8)

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Exhibit "C"
After 30 Days, Run 3

Exhibit "D"
After 60 Days, Run 3B

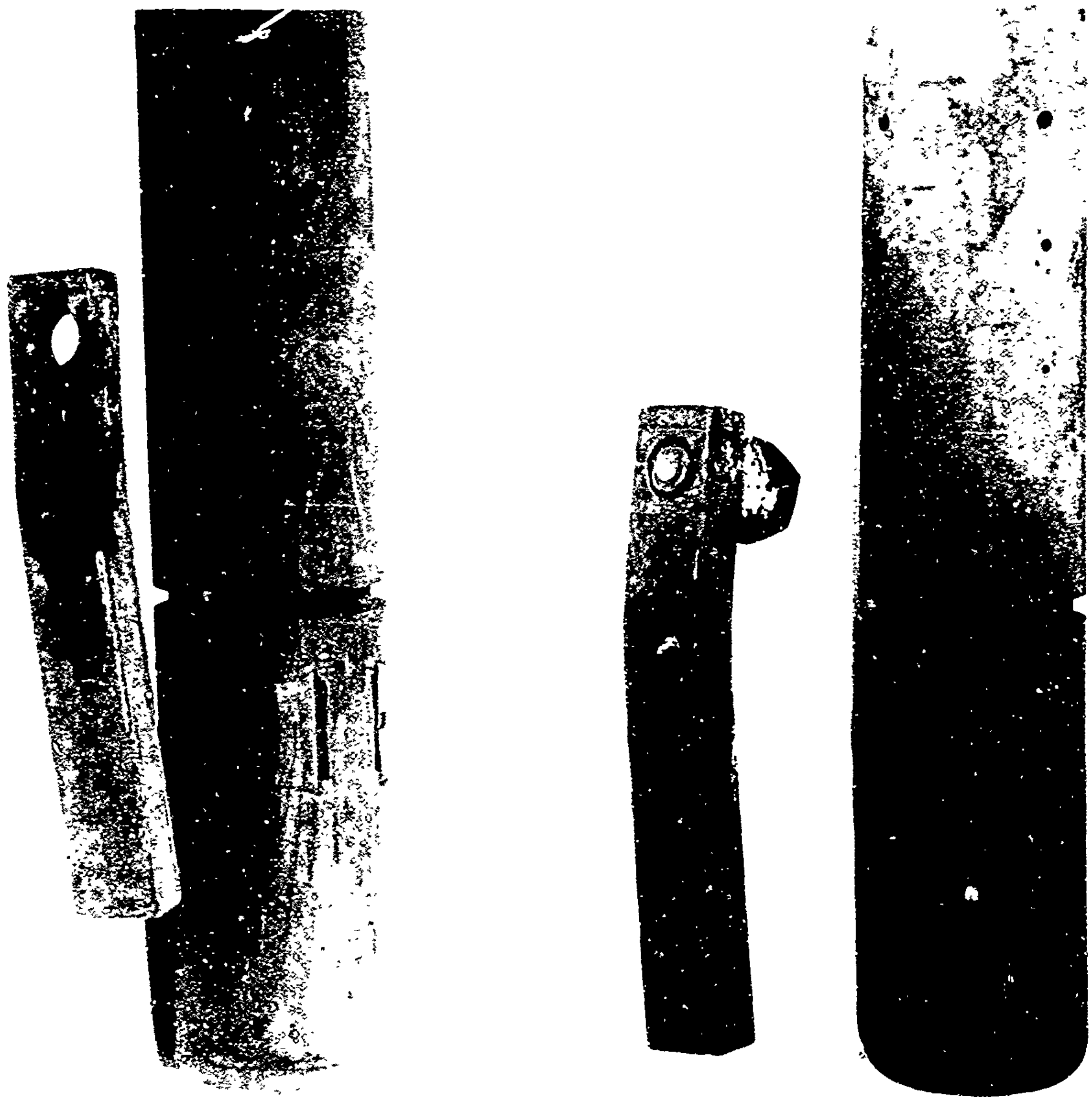

Figure 2
Boiler Tubes and Embrittlement Specimen After Exposure to 1200 PSI Boiler Water Treated With LiOH and Na_2 EDTA

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Figure 3
Photomicrograph, Example of Typical Surface Condition of Boiler Tube Metal (Wall of 1/8-Inch-Diameter Hole) After 30-Day Exposure to NaOH Treated Water (Run 1) (250X)

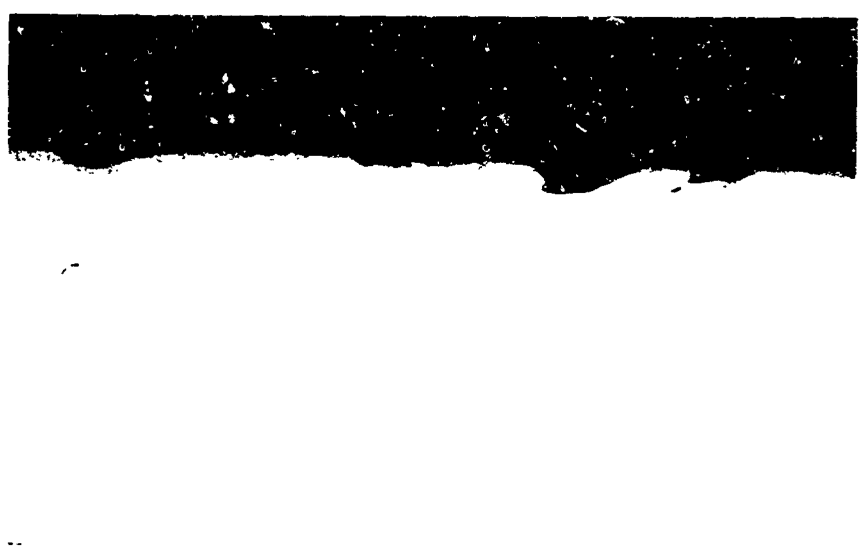

Figure 4
Photomicrograph, Example of Typical Surface Condition of Boiler Tube Metal (Wall of 1/8-Inch-Diameter Hole) After 30-Day Exposure to LiOH Treated Water (Run 2) (250X)

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